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**NASA-SPONSORED
CONTAINERLESS PROCESSING
EXPERIMENTS**

Prepared for

**The Workshop on Containerless
Processing in Microgravity**

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OUTLINE

GROUND BASED EXPERIMENTS

DROP TUBE STATUS
PYROMETRY
SOLIDIFICATION VELOCITY

EM LEVITATION
FACILITIES
EXPERIMENTS

AERODYNAMIC LEVITATION

ACOUSTIC LEVITATION

PURIFICATION

IML-2 MISSION

COMPARISON OF CONTAINERLESS PROCESSING TECHNIQUES FOR BULK SAMPLES

| | MSFC TUBE | UHV TUBE | EM in LAB | TEMPUS |
|--------------------------|---|--------------------------------------|------------------------|-------------------------------|
| Environmental purity | fair | excellent | excellent | good |
| Vacuum (torr) | 10^{-5} - 10^{-6} | 10^{-8} - 10^{-10} | 10^{-9} - 10^{-10} | 10^{-9} |
| Experimental duration | 4.6 sec | 3.1 sec | unlimited | flight timeline limited |
| Quiescence | good | good | large agitation | small agitation |
| Temp. measurement | will improve with drop tube pyrometry | | excellent | potentially excellent |
| Temp. control | radiation cooling rate is slowest, can be varied with gas pressure | | good | best |
| Isothermality | poor | poor | okay | okay |
| Position control | free fall | | stability problems | excellent |
| Accessability | good | presently only French facility | excellent | poor |

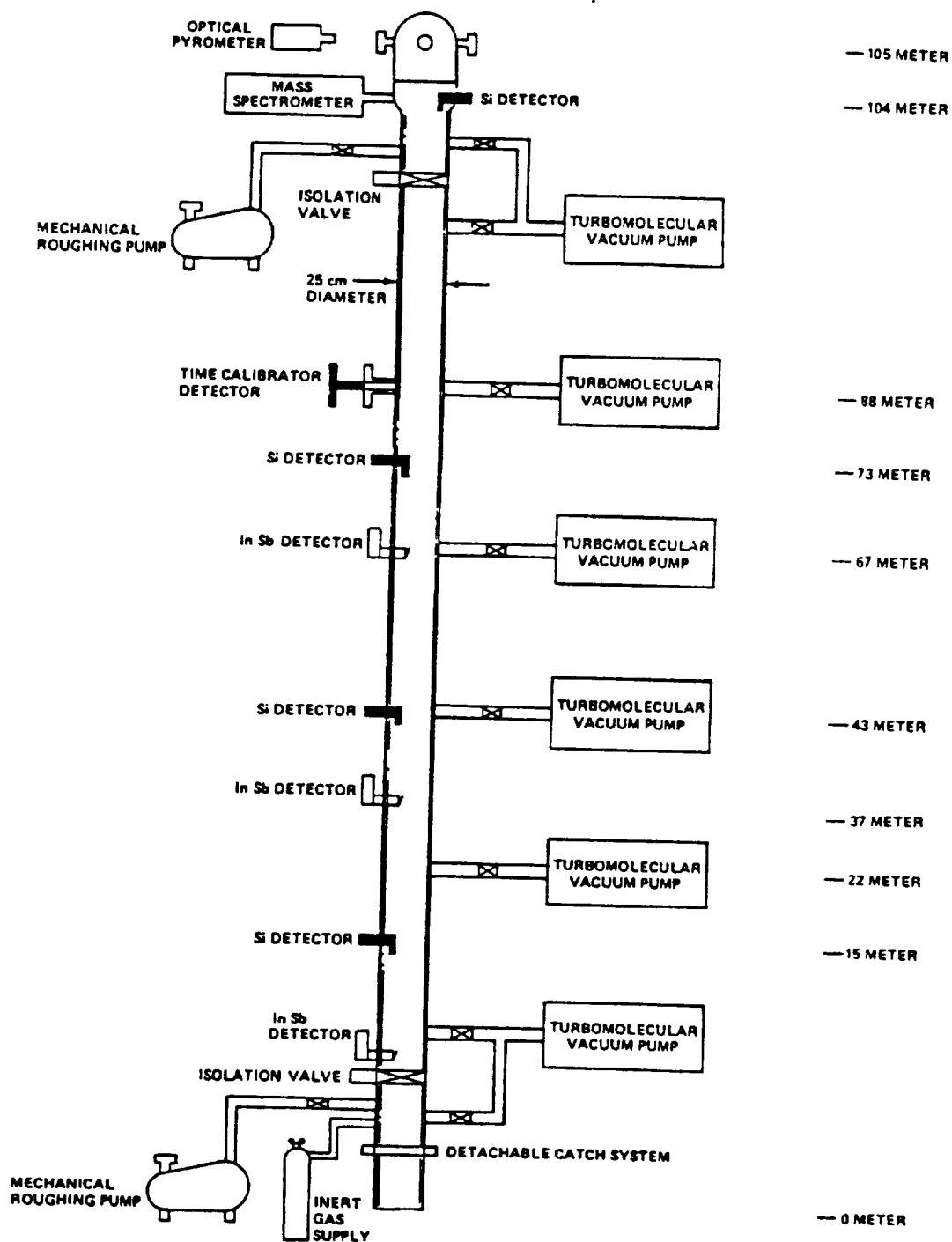
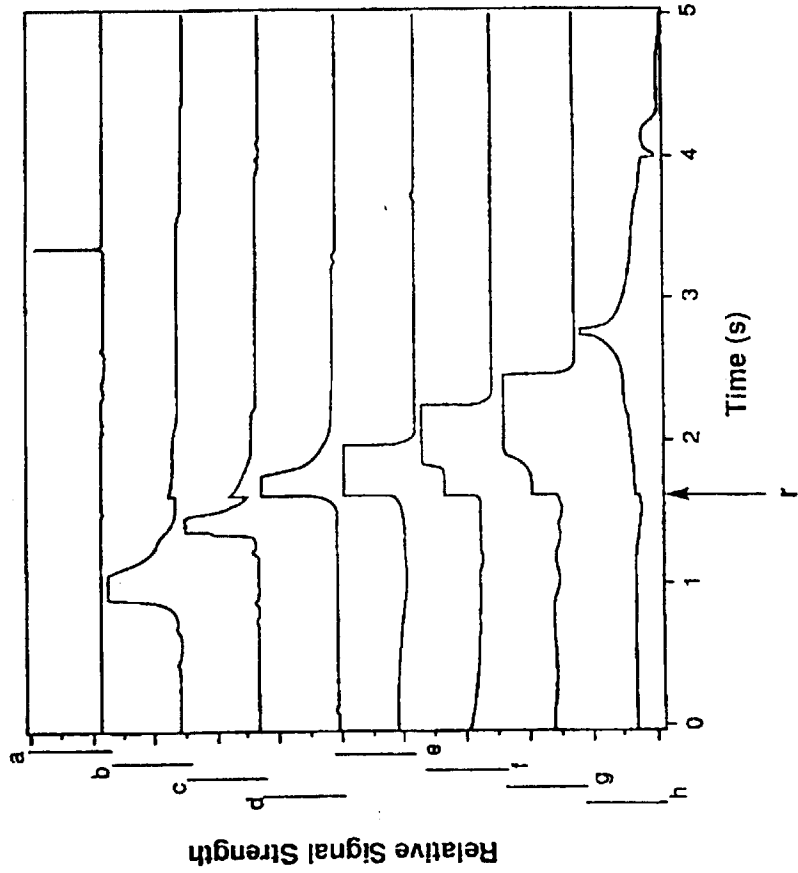
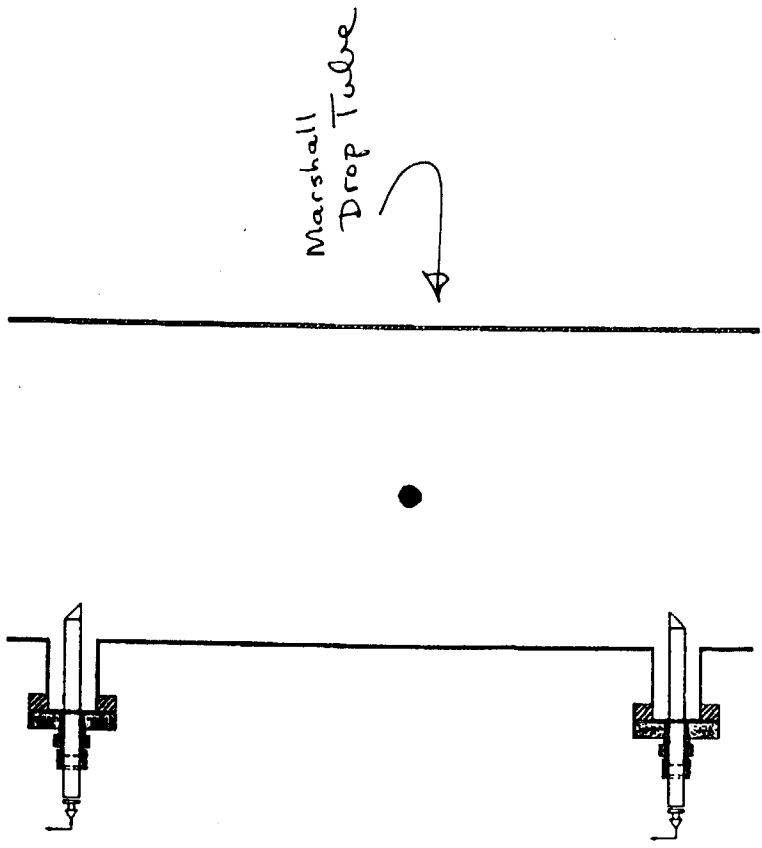
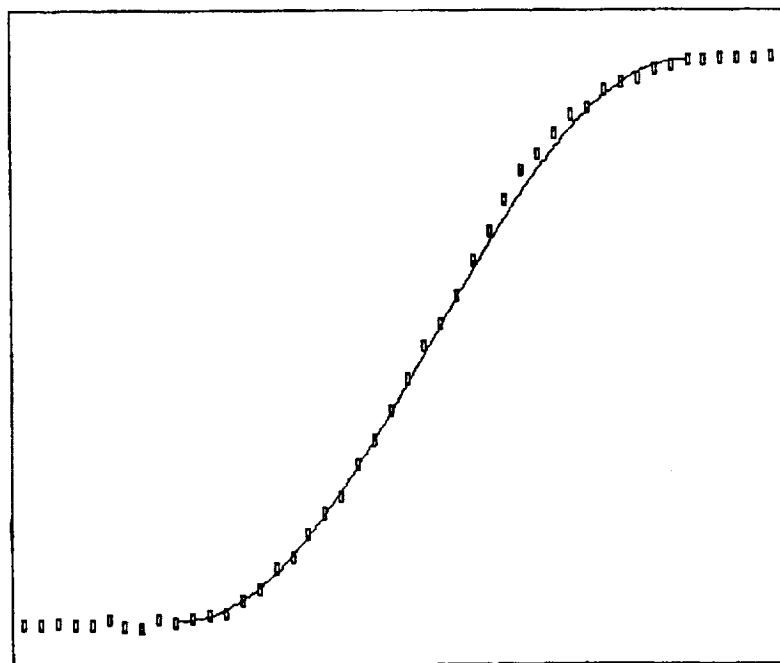
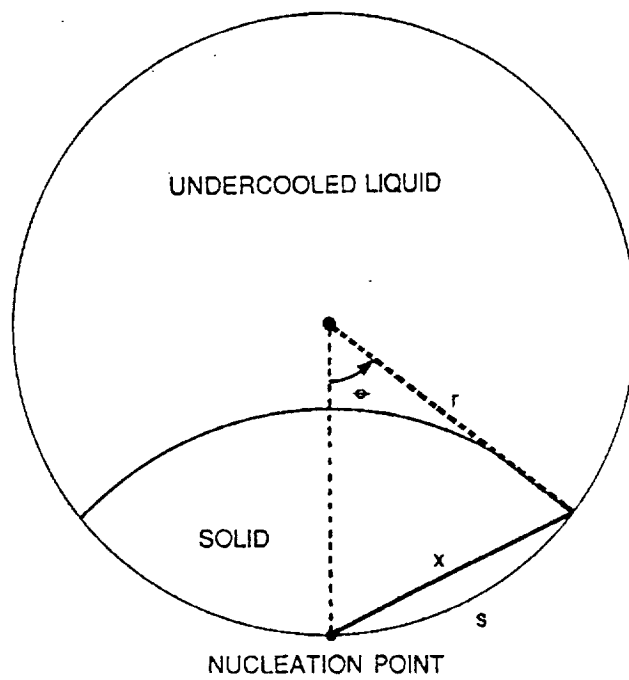


Figure 1. Schematic of the 105-meter drop tube at Marshall Space Flight Center.



Legend:

- a = Timer Detector on level 5.
- b, c, d = Down-Looking Detectors on levels 14, 13, 12.
- e, f, g = Up-Looking Detectors on levels 11, 10, 9.
- h = Up & Down-Looking Logarithmic Amplifier/Detector on level 8.
- r = Recalescence Event



MEASURED SOLIDIFICATION VELOCITIES IN MSFC DROP TUBE

| PURE Nb | Nb-Pt (Primary Nb) | Nb-Pt (Primary Nb ₃ Pt) |
|---------|--------------------|------------------------------------|
| 20 m/s | 15 m/s | 1 m/s |

Arc Cast

Ti - Ce Phase Diagram

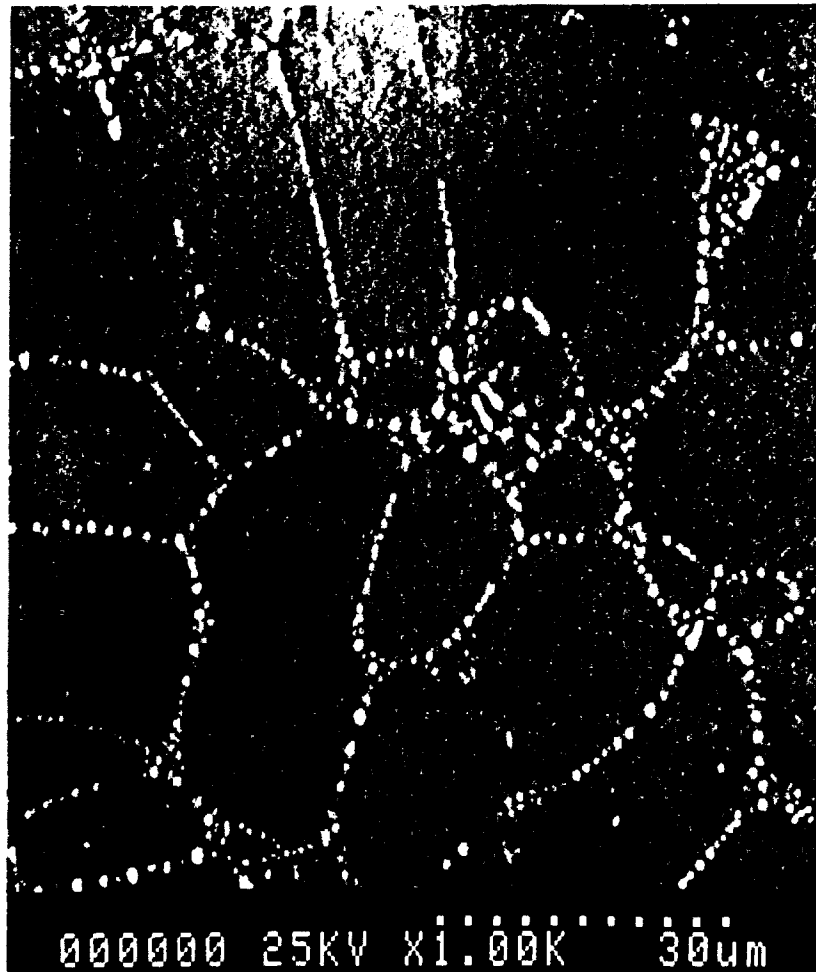


FIGURE 1. SEM OF THE ARC CAST ALLOY SHOWS MASSIVE SEGREGATION OF CERIUM AT THE PRIOR BETA TITANIUM GRAIN BOUNDARIES.

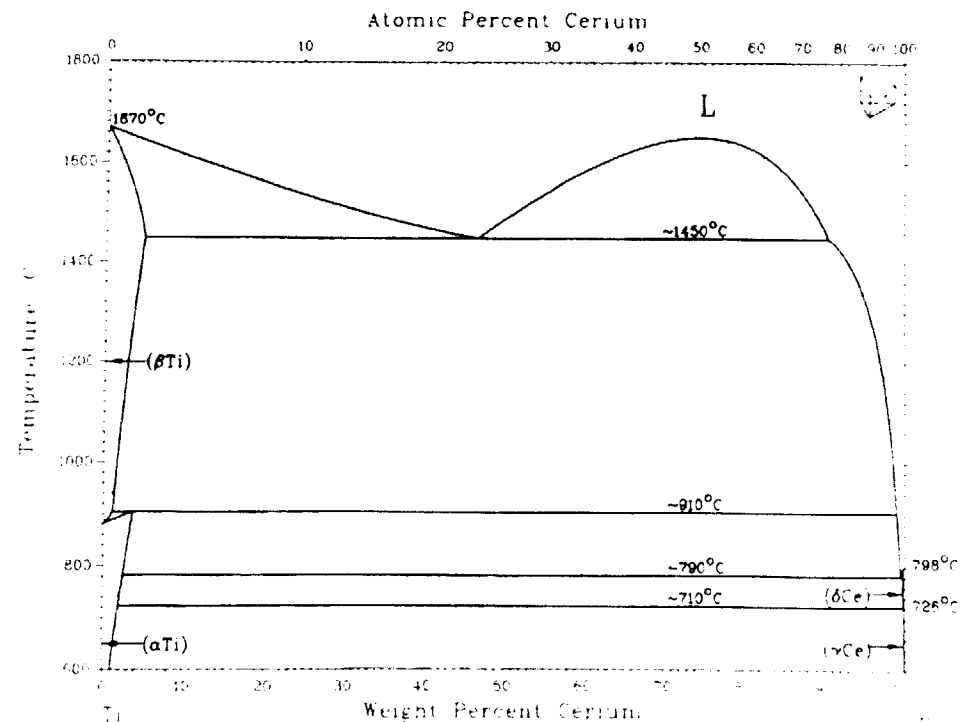


FIGURE 2. THE TITANIUM-CERIUM BINARY EQUILIBRIUM PHASE DIAGRAM REVEALS A MONOTECTIC IMMISCIBILITY GAP IN THIS SYSTEM.

Drop Tube - Gas Cooled

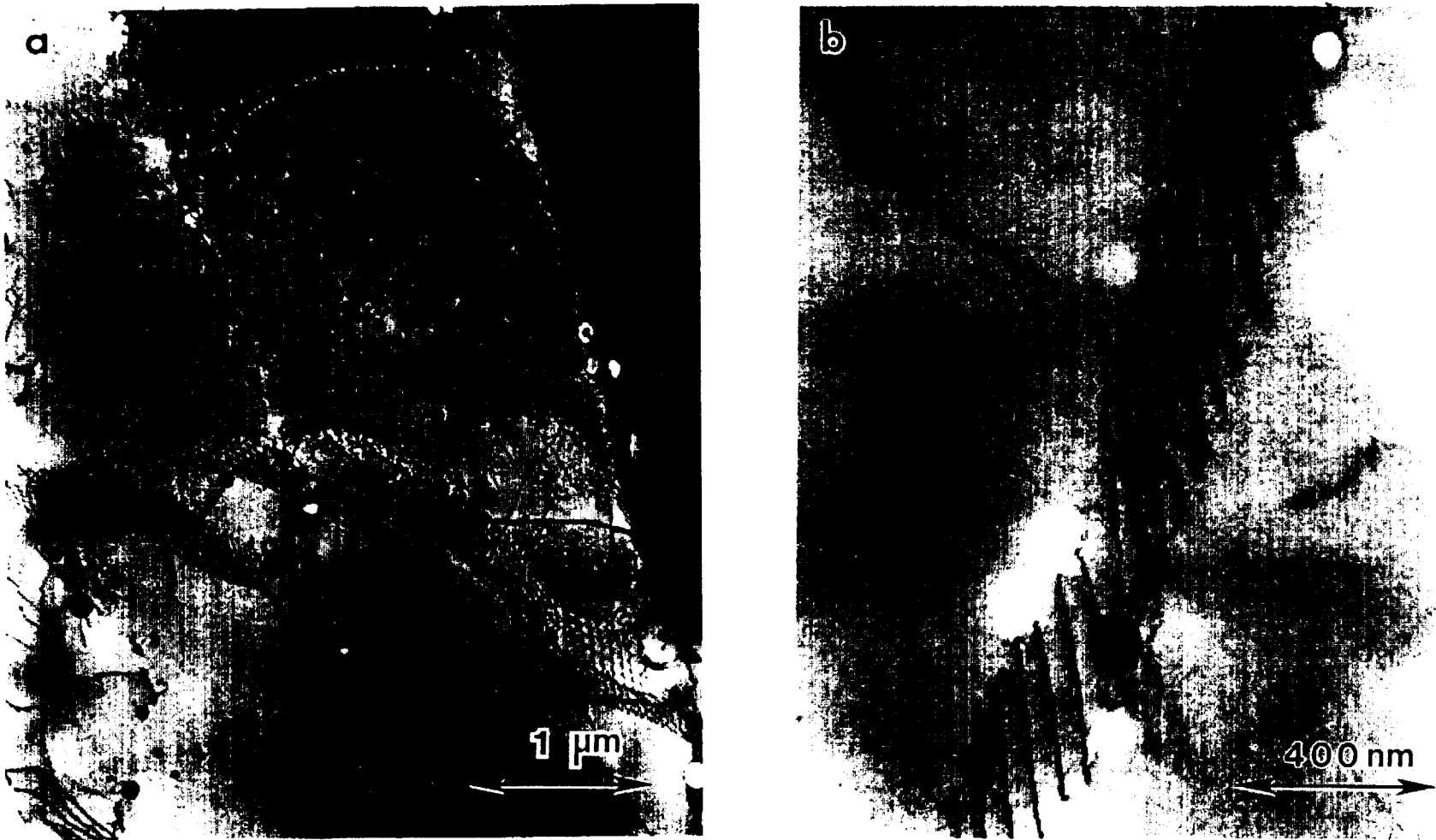


FIGURE 3. THE BRIGHT FIELD TEM IMAGES IN (a) AND (b) OF THE MICROSTRUCTURE FROM THE GAS COOLED DROP TUBE SAMPLES REVEAL AN ALPHA HEXAGONAL TITANIUM MATRIX WITH NUMEROUS LOW ANGLE GRAIN BOUNDARIES THAT ARE DECORATED WITH CERIUM PRECIPITATES. WITHIN THE ALPHA TITANIUM MATRIX, PRECIPITATION OF CERIUM IS ABSENT.

Undercooled and Rapidly Solidified

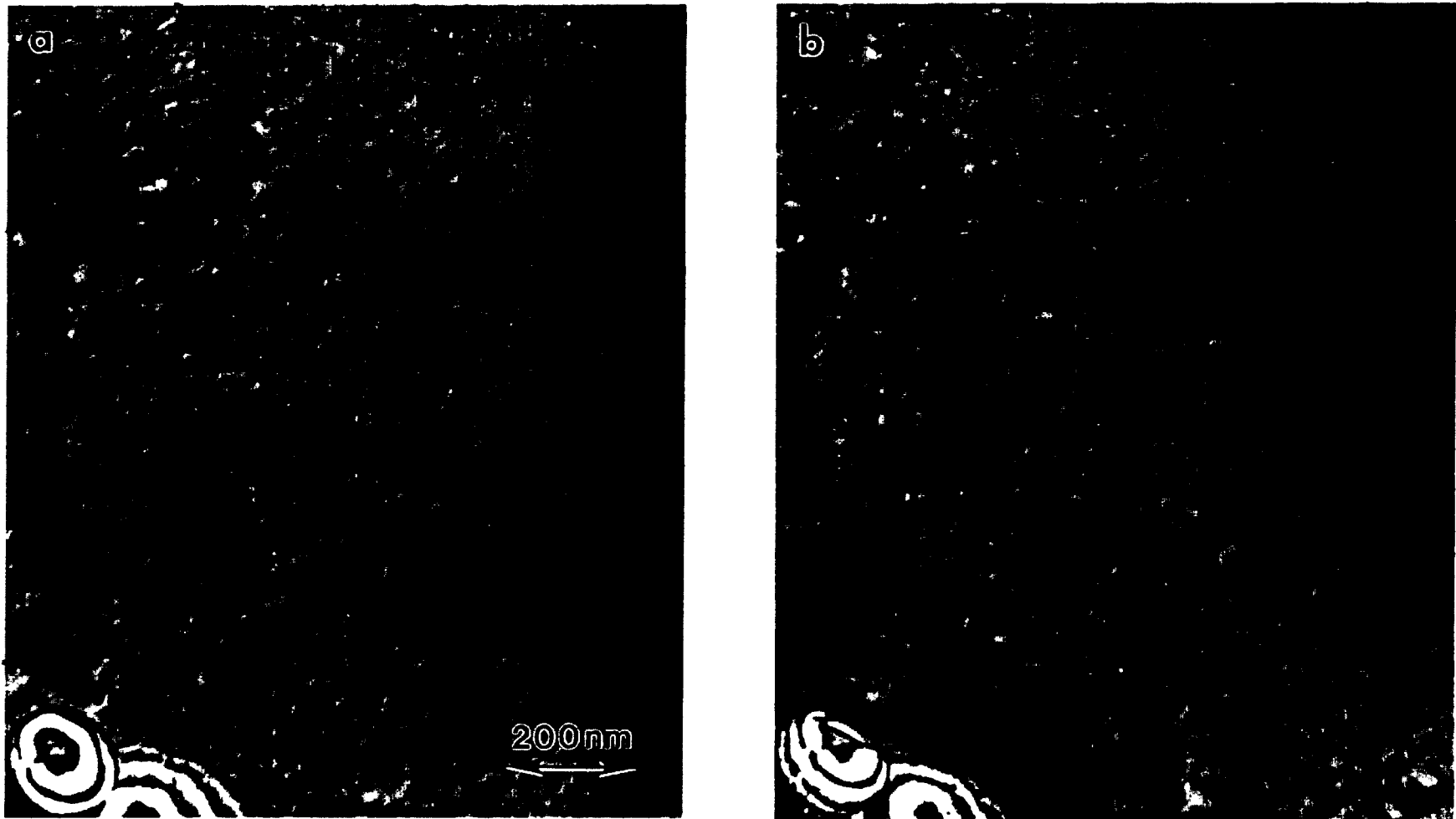
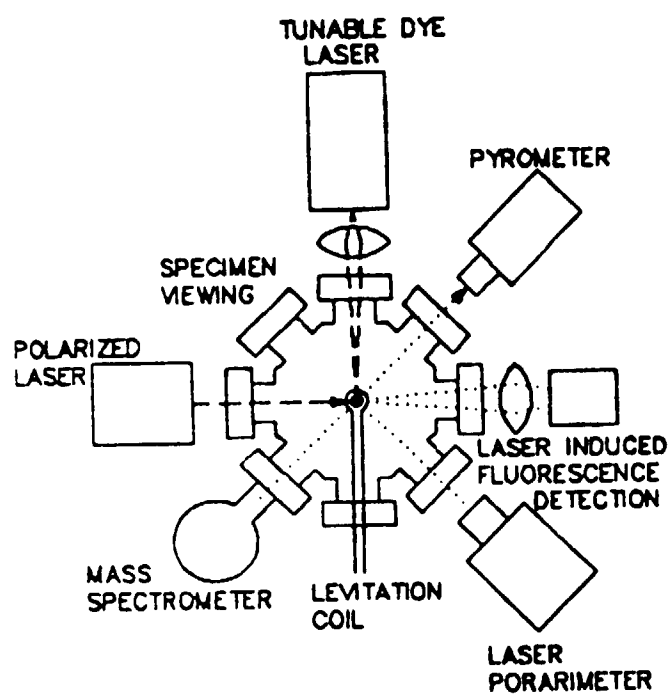


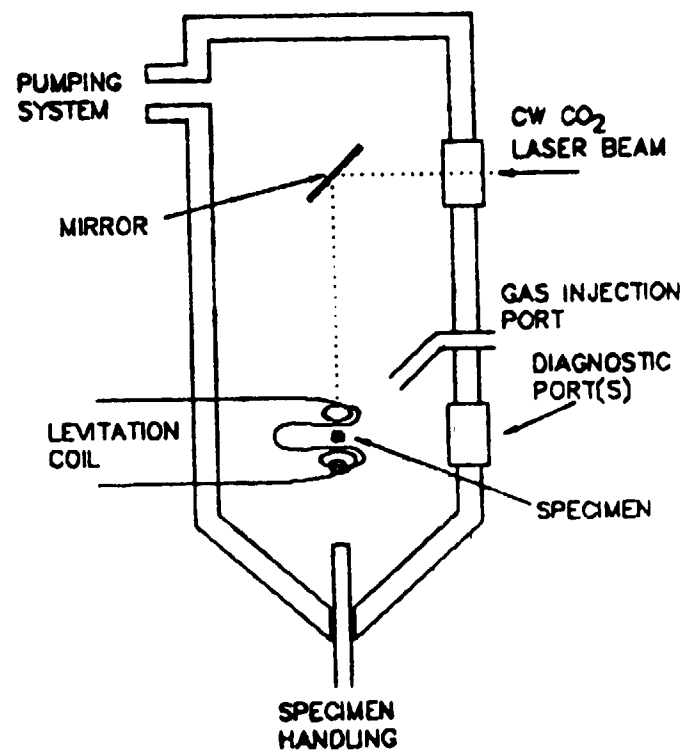
FIGURE 4. TEM EXAMINATION OF THE UNDERCOOLED AND RAPIDLY SOLIDIFIED MICROSTRUCTURE REVEALS A FINE DISTRIBUTION OF PARTICLES IN THE ALPHA TITANIUM MATRIX. THE PARTICLES, WHICH ARE 5 TO 50 nm IN SIZE, APPEAR TO BE RANDOMLY DISTRIBUTED IN (a). HOWEVER, TILTING THE SAME REGION OF THE SAMPLE IN (b) INDICATES THAT THE PARTICLES HAVE FORMED IN LAYERS DURING THE BETA TO ALPHA TRANSFORMATION.

SUMMARY OF CONTAINERLESS PROCESSING FACILITIES AT INTERSONICS

- * Electromagnetic, acoustic and aerodynamic levitation
- * Laser beam and arc lamp heating systems
- * State of the art non-contact temperature and optical property measurement facilities
- * Non-intrusive diagnostic techniques with LIF and mass spectrometer
- * Controlled atmosphere processing
- * Gas quenching
- * Proven microgravity processing technology



PLAN VIEW

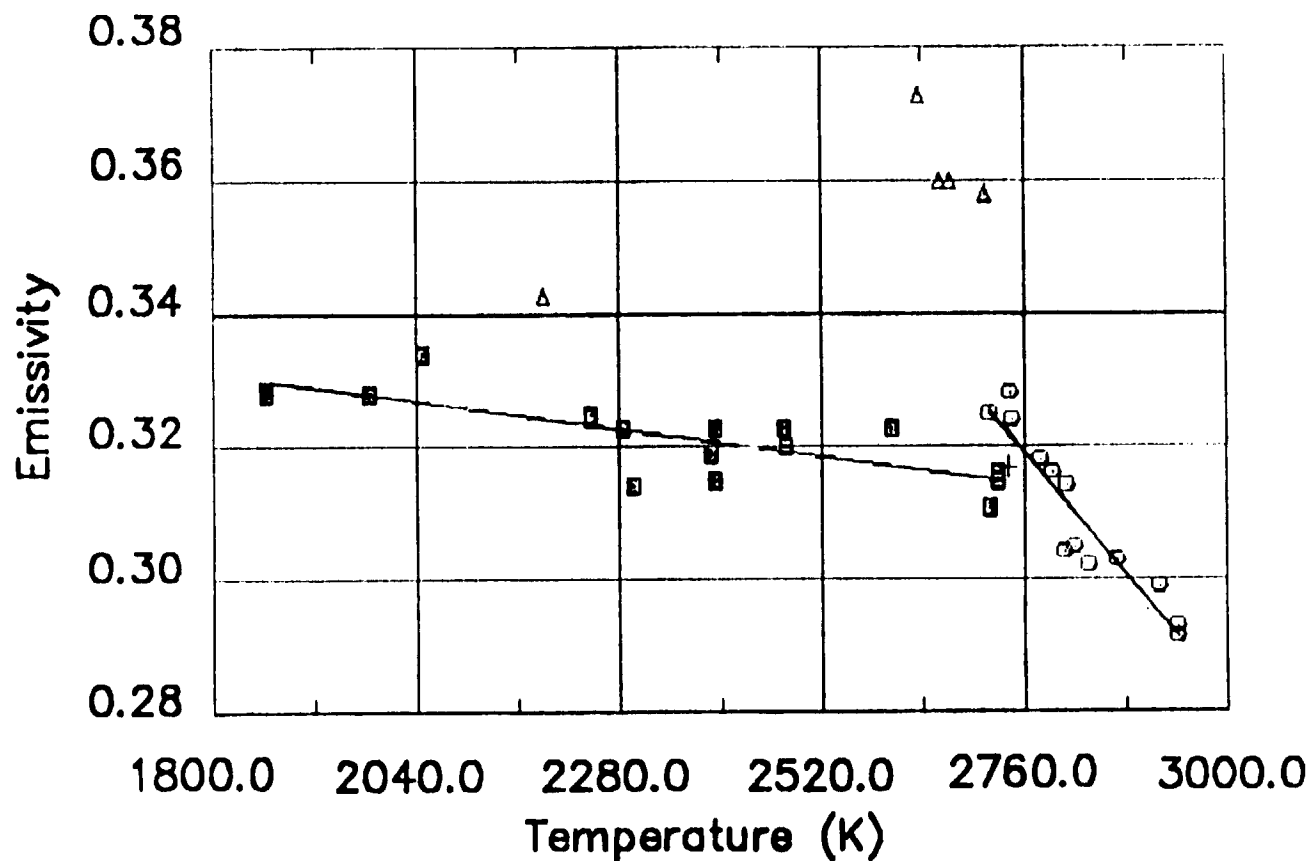


ELEVATION

INTERSONICS GROUND-BASED ELECTROMAGNETIC LEVITATOR



The normal spectral emissivity of Niobium as a function of temperature at $0.6328 \mu\text{m}$.



○ – Liquid Niobium ■ – Solid Niobium ▲ – Solid Niobium before clean up
+ – Values at T_m and $0.65 \mu\text{m}$ (D. W. Bonnell)

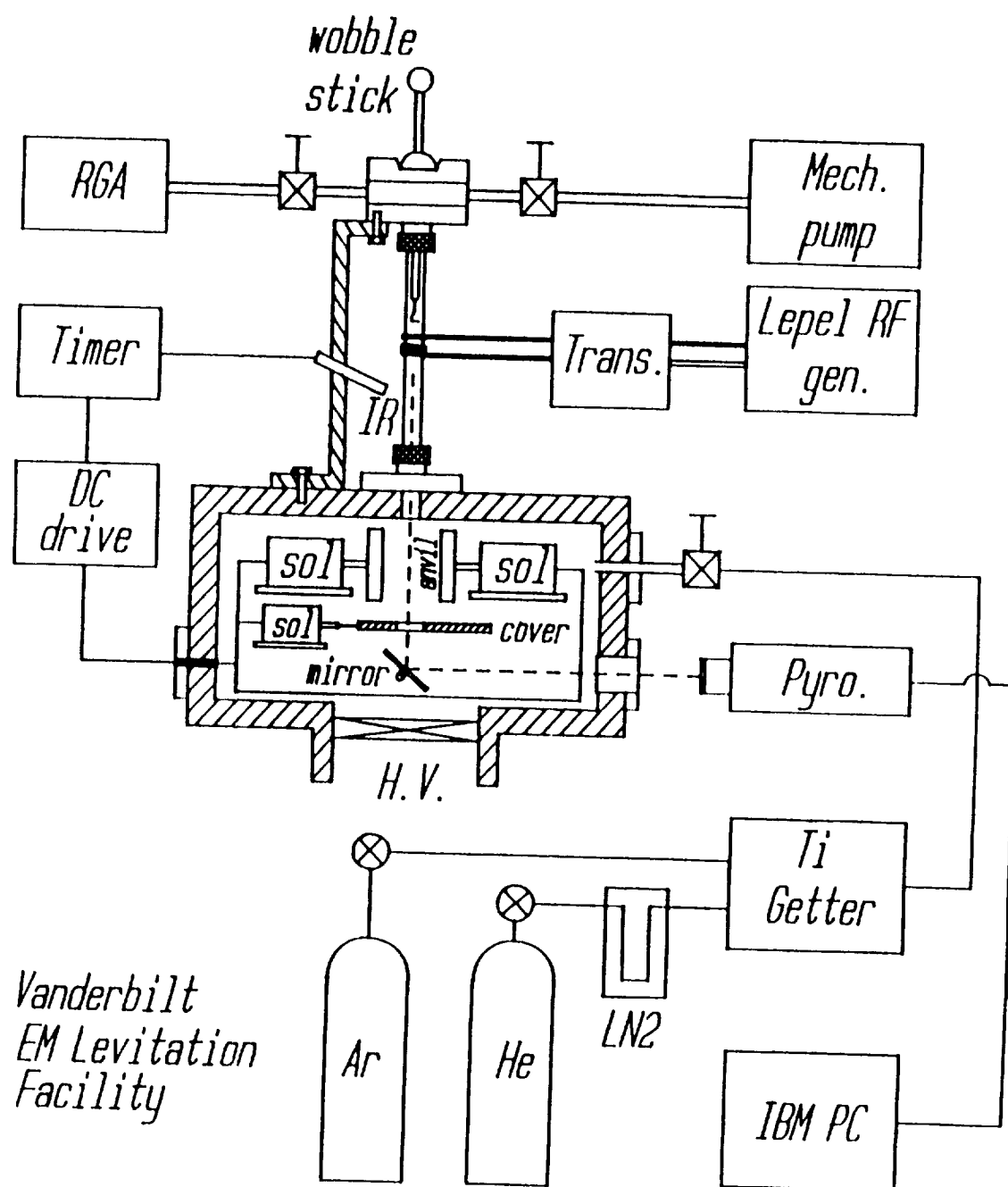
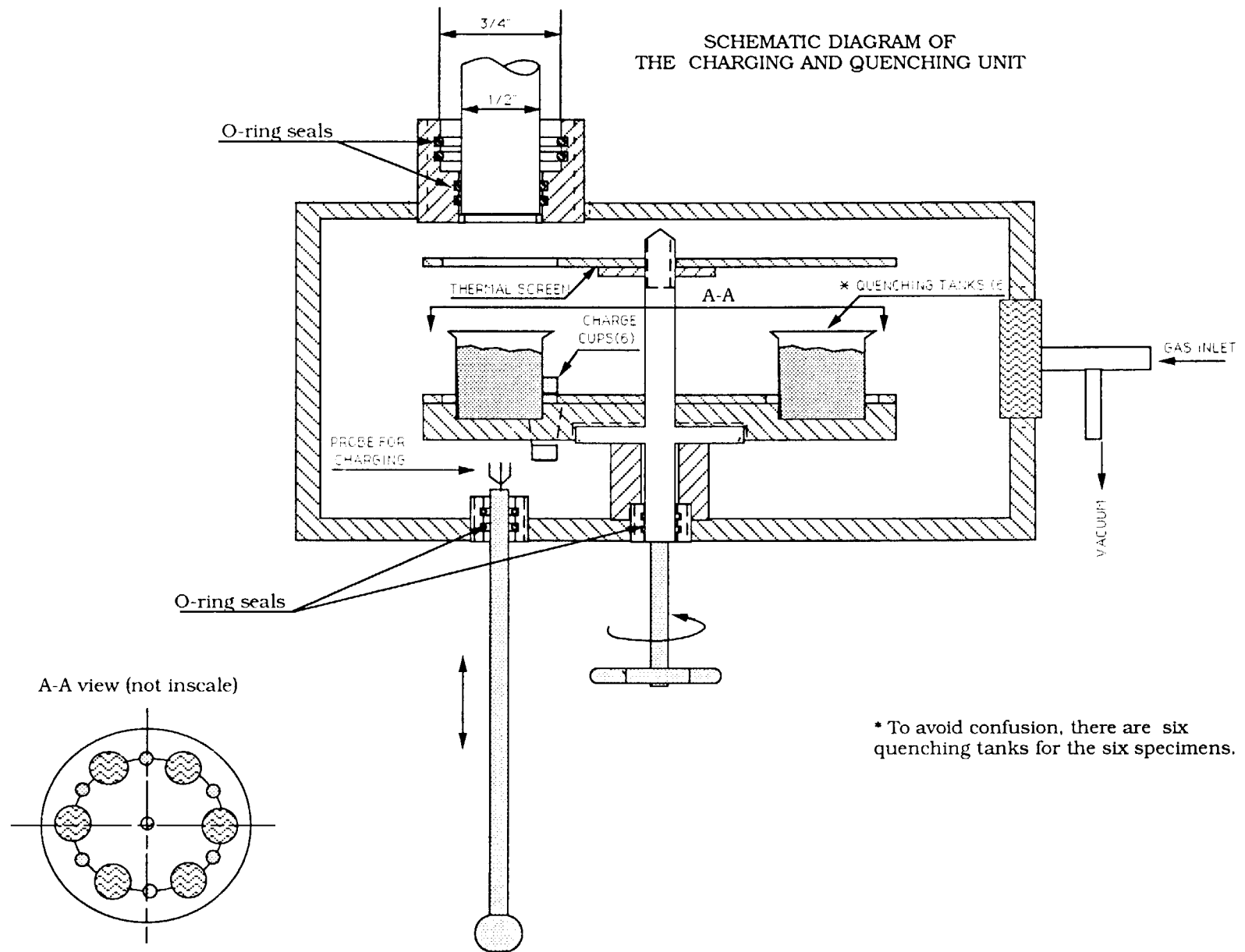
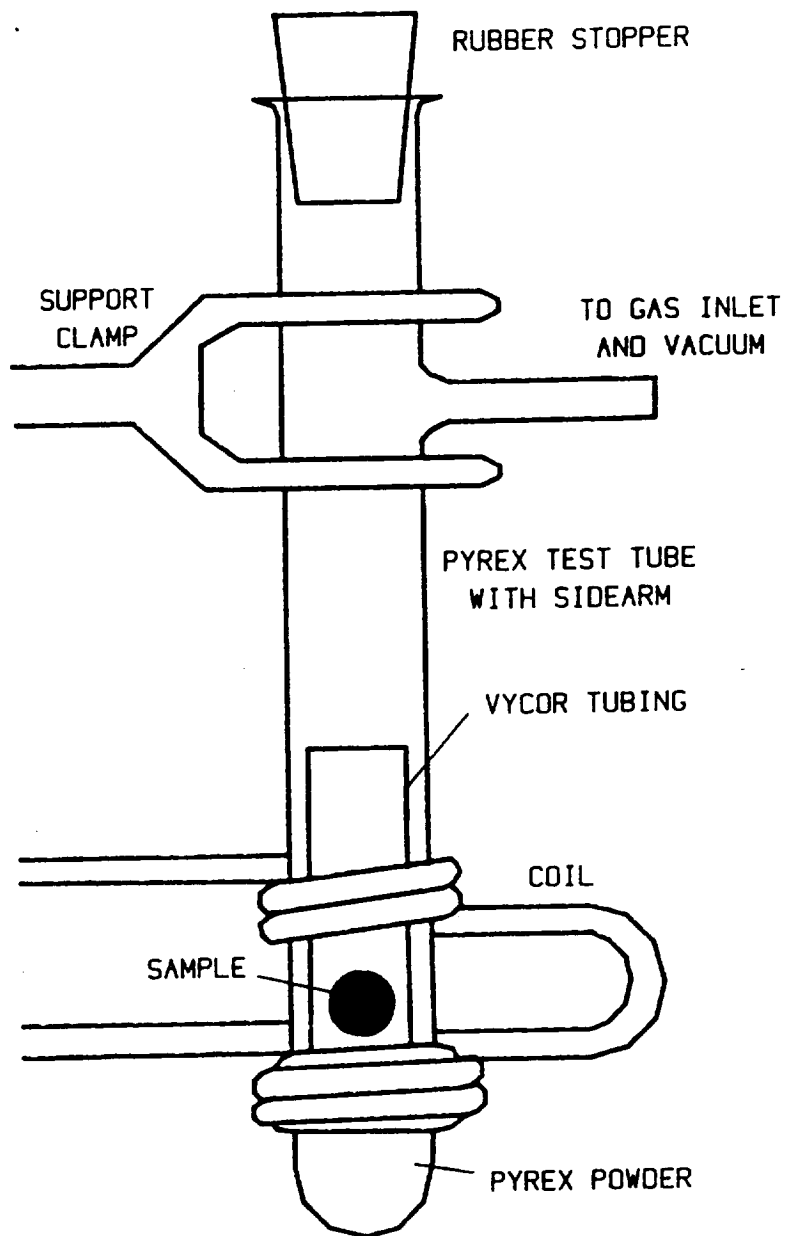


Figure 5. Schematic of the Electromagnetic Levitation Facility at Vanderbilt University.

SCHEMATIC DIAGRAM OF THE CHARGING AND QUENCHING UNIT





SUMMARY OF RESULTS
FOR
EM LEVITATION
OF Nb 16 TO 22 At.% Si ALLOYS

EUTECTIC RANGE

Metallic glass formation observed only on superheated and splatted samples.

Undercooled and splatted samples had extremely fine regular and irregular eutectic microstructures.

Undercooled gas quenched samples solidified with microstructures identical to drop tube counterparts.

Nb₃Si RANGE

Nb₃Si growth directly from the liquid for a wide composition range in splatted samples.

The depth to which this solidification path is followed depends on the previous bulk undercooling.

Undercooled and gas quenched samples tend to solidify with primary Nb₅Si₃ and a metastable α -Nb + Nb₅Si₃ eutectic. The peritectic Nb₃Si does not form.

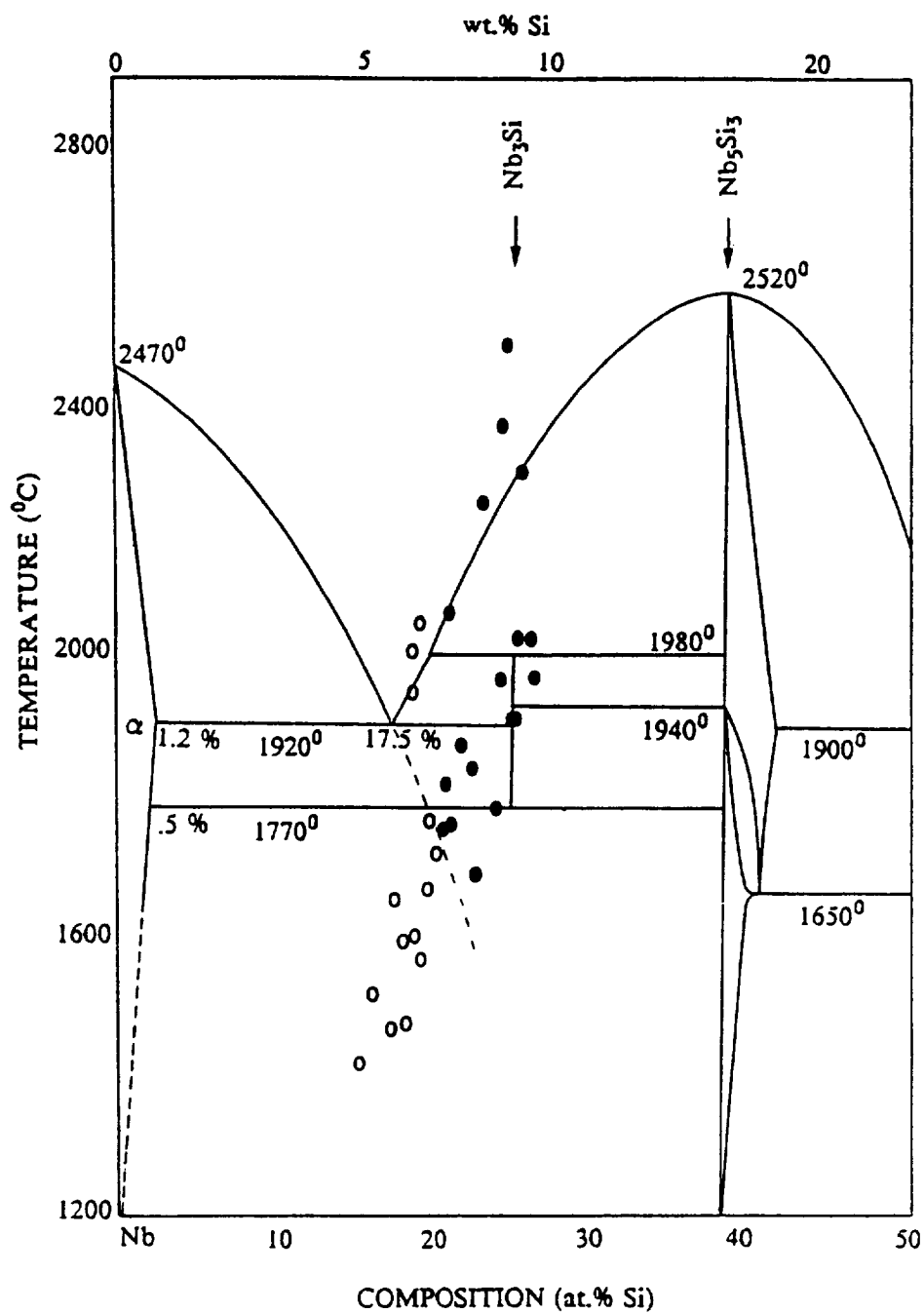
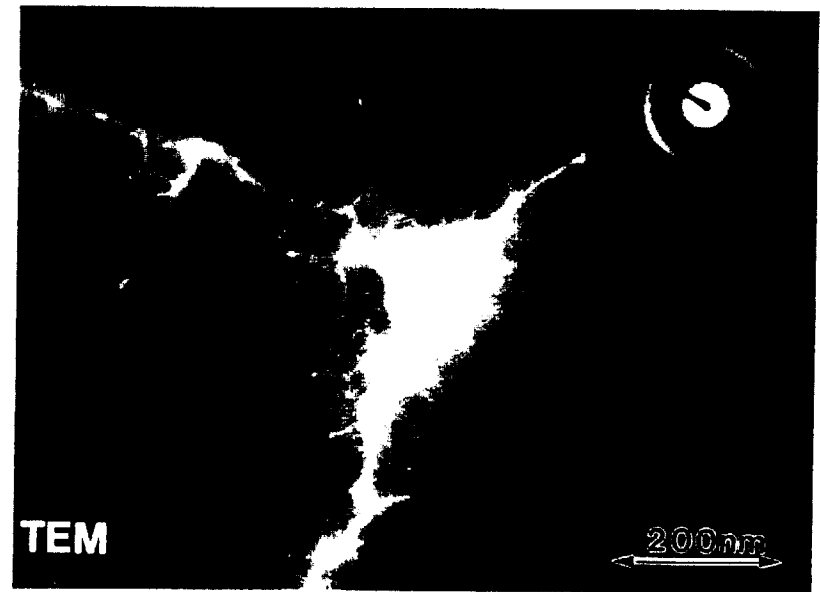
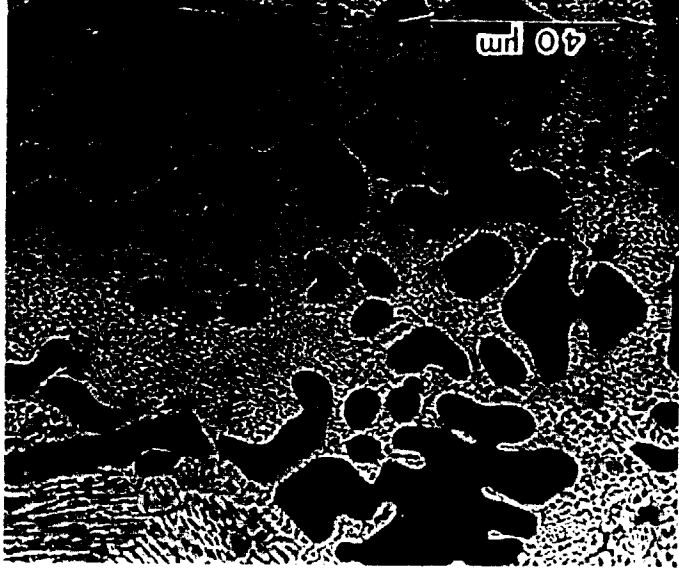
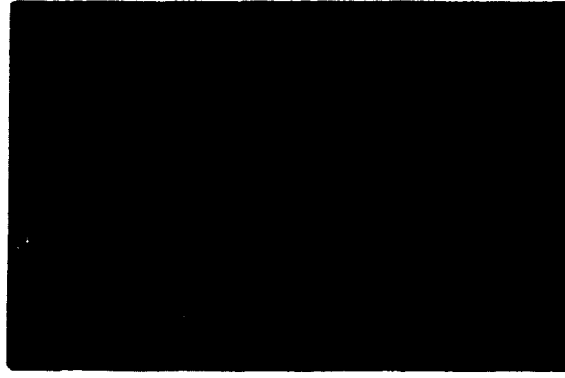
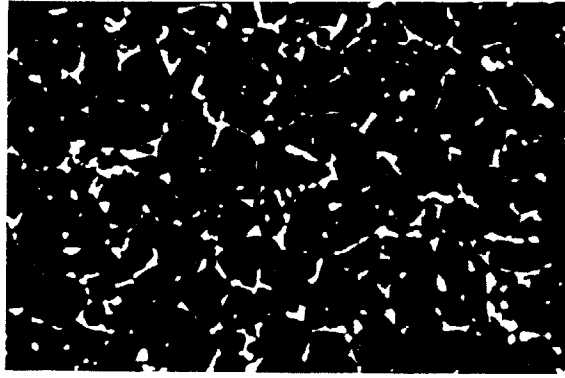
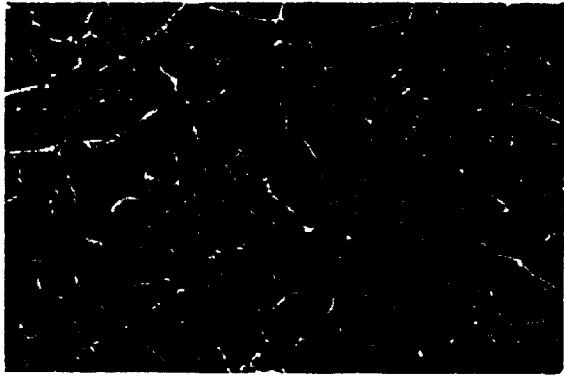


Figure 6. Nb-Si phase diagram. Empty symbols on the phase diagram represent undercooling prior to splatting and composition of samples that solidified without primary phases. Solid symbols represent those samples that solidified with primary phases.

Nb 16 TO 20 At.% Si



Nb 22 TO 27 At.% Si



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